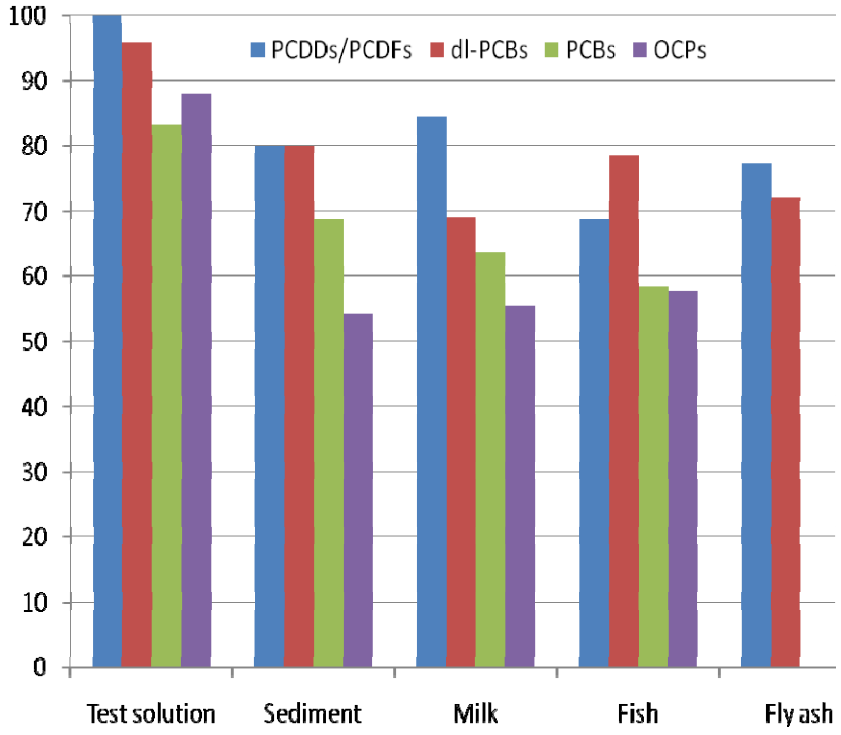
First Worldwide UNEP Intercalibration Study on Persistent Organic Pollutants - Asia Region



United Nations Environment Programme

Division of Division of Technology, Industry, and Economics
Chemicals Branch

June 2010

IOMC

INTER-ORGANIZATION PROGRAMME FOR THE SOUND MANAGEMENT OF CHEMICALS

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Acronyms and Abbreviations

AV	Assigned value
dl-PCB	Dioxin-like polychlorinated biphenyls
dl-POPs	Dioxin-like POPs
GC	Gas chromatograph(y)
GPC	Gel permeation chromatography
HCB	Hexachlorobenzene
LCV	Left-censored values (values below detection limit)
MS	Mass spectrometer
MSWI	Municipal waste incinerator
NA	Not applicable
ND	Not detected
OCP	Organochlorine pesticide
PCB	Polychlorinated biphenyl
PCDD/PCDF	Polychlorinated dibenzo-*para*-dioxins/polychlorinated dibenzofurans
PDF	Probability density function
PMF	Main mode
POPs	Persistent organic pollutants
RSD	Relative standard deviation
SD	Standard deviation
TCDD	2,3,7,8-Tetrachloro-*p*-dibenzodioxin
TEQ	Toxic equivalent
TEQ_{PCB}	Toxic equivalent based on dl-PCB
$TEQ_{PCDD/PCDF}$	Toxic equivalent based on PCDD and PCDF (dl-PCB not included)
TEQ_{total}	Toxic equivalent based on PCDD, PCDF, and dl-PCB
WEOG	Western European and Other Groups

Definitions

Basic POPs	Include: Organochlorine pesticides (aldrin, chlordane, DDT, dieldrin, endrin, heptachlor, hexachlorobenzene, mirex, toxaphene) and polychlorinated biphenyls
Dioxin-like POPs	Include 29 congeners that were assigned a TEF by WHO/IPCS expert group, namely polychlorinated dibenzo-*para*-dioxins, polychlorinated dibenzofurans, and polychlorinated biphenyls

Summary

The first worldwide interlaboratory study on persistent organic pollutants (POPs) under the Stockholm Convention was organised in the Asian/Pacific region. In addition to Asian laboratories, several laboratories from OECD countries participated. The participating laboratories had a choice to analyse different matrices: two test solutions, a fish sample, a sediment, a fly ash and a human milk sample. In total, 38 laboratories from Asia, Europe and North America participated in the present study. Of these, 35 laboratories submitted data on the test solutions (either for basic POPs or for dioxin-like POPs), 30 on the sediment, 20 on the human milk, 24 on the fish and 24 on fly ash. All results were statistically evaluated according to the procedures used in the QUASIMEME proficiency testing scheme.

The best results were obtained for the test solutions, and, as regards to chemical compounds, for PCDD/PCDF. The fish sample was the most difficult matrix for the participants as were the OCP as a compound class. In general the results for the dioxin and dioxin-like PCB were good and in agreement with and in some cases better than reported for this complex analysis. The uncorrected $TEQ_{PCDD/PCDF}$ values had an RSD of 29 % for the human milk sample, whereas the uncorrected RSDs for OCP and some PCB were often higher than 100 %. This result was also true for the fish matrix, in which the $TEQ_{PCDD/PCDF}$ showed an RSD of 33 %. For indicator PCB and OCP results were comparable to an earlier, smaller interlab study organised by a UNEP/GEF project for the Stockholm Convention during a pilot project on capacity building.

This intercalibration study showed that further analytical improvement is required for especially organochlorine pesticides and PCB since analysis of the core matrices in the Global Monitoring Programme under the Stockholm Convention, human milk and air samples, are even more demanding than those used in this study. This emphasises the need for all laboratories to pay more attention to quality assurance (QA) and method development. Clearly, the use of labelled standards and GC/MS is a key factor in decreasing variation between the laboratories, as is shown for the PCDD/PCDF results.

Somewhat surprising is the fact that a larger number of laboratories reported data for the PCDD/PCDF and dioxin-like PCB than for the marker PCB and organochlorine pesticides (except for DDTs in sediment). The analysis of pesticides is generally considered to be less complicated and less complex instrumentation is needed. However, among the 38 laboratories, only very few used low resolution GC/MS systems or GC/ECD. Several participants represent national expert laboratories with access to high resolution GC/ high resolution MS systems optimised for dioxin analysis. This specialization might explain the relatively large variation observed for the pesticide analysis where all types of detectors were used (ECD, LRMS, and HRMS).

1. Introduction

Within the framework of the United Nations Environment Programme's (UNEP) Capacity Building project for training of laboratory staff on persistent organic pollutants (POP) analysis in developing countries, the Institute for Environmental Studies of the VU University Amsterdam, The Netherlands (IVM) and the MTM Research Center, School of Science and Technology at the University of Örebro, Sweden, have organised the First Worldwide UNEP Interlaboratory Study on Persistent Organic Pollutants (POPs). The first phase of this study was financed by the Norwegian Government and was conducted in Asia. In addition to the developing countries from Asia-Pacific, POPs laboratories from developed countries were invited to participate as wel. The results of the study are presented in this report. The POPs studied included polychlorinated-*p*-dibenzodioxins (PCDD), polychlorinated dibenzofurans (PCDF), polychlorinated biphenyls (PCB) and the organochlorine pesticides (OCP), *i.e.,* DDT and metabolites, mirex, dieldrin, endrin, aldrin, chlordanes, hexachlorobenzene, heptachlor and *cis*-heptrachlorepoxide. Toxaphene was not included since no or only limited capacity was available among the participating laboratories.

In total, five matrices were offered for analysis: Standard solutions for POPs pesticides, for indicator PCB, and for dioxin-like POPs, sediment, fish, fly ash (for dioxin-like POPs only), and human milk. The test solutions in amber glass ampoules with the target compounds in unknown concentrations were sent to the participating laboratories. The sediment was air-dried, the fish consisted of a freeze-dried samples, and the human milk was homogenised and frozen before shipment.

Thirty eight laboratories from 13 countries participated (see Appendix 1 for their names and addresses as well as the abbreviations (codes) that have been used throughout this report). All codes are confidential and are only revealed to third parties after permission of the participant.

In the following chapters the results of the study will be discussed. The final chapter covers general conclusions and recommendations.

2. Materials and Methods

2.1 Preparation of the test samples

The ash sample was a fly ash sample from a MSWI incinerator from Sweden taken after the bag house filter and wet scrubber. The ash was used as received (dry) and homogenised at the MTM Research Center at the Örebro University. The ash contained medium levels of the target compounds except for the pesticides which were not present in this ash sample.

The sediment originates from Norway and was air-dried at 40 °C and sieved (0.5 mm pore size). After homogenisation, individual plastic containers were filled with the test matrix and stored at room temperature until shipment.

The fish sample consists of a freeze-dried fish sample from the Great Lakes, made available by Dr. Eric Reiner from the Ontario Ministry of Environment, Laboratory Services Branch, Ontario, Canada.

The human milk sample consisted of pooled, homogenised milk from the Swedish mother milk bank in the Stockholm area. The milk samples were frozen and stored at -20 °C before shipment.

Standard 1A consisted of a mixture of PCDD/PCDF and dl-PCB in the concentration range from 10 pg/µl to 500 pg/µl (ng/ml). This standard was prepared, ampouled and labelled by Wellington Laboratories (Guelph, Ontario, Canada).

Standard 1B consisted of a mixture of the indicator PCB (PCB 28, 52, 101, 138, 153 and 180) in the concentration range from 0.1 ng/µl to 5 ng/µl (µg/ml). This standard was prepared, put into ampoules and labelled by Wellington Laboratories (Guelph, Ontario, Canada).

Standard 1C consisted of a mixture of organochlorine pesticides (OCP) in the concentration range from 10 pg/µl to 50 pg/µl (ng/ml). This standard was prepared by IVM from a standard solution obtained from Cambridge Isotope Laboratories (Andover, USA). After preparation, the aliquots were ampouled, labelled and stored at room temperature. The OCP present in the solution were HCB, aldrin, dieldrin, endrin, *p,p'*-DDT, *p,p'*-DDE, *p,p'*-DDD, *o,p'*-DDT, *o,p'*-DDE, *o,p'*-DDD, *trans*-chlordane (*gamma*), *cis*-chlordane (*alpha*), *trans*-nonachlor, *cis*-nonachlor, oxychlordane, heptachlor, *trans*- heptachloroepoxide (HEPO), *cis*-HEPO, mirex, α-HCH, β-HCH, γ-HCH, δ-HCH. Although present, the HCHs were not part of the study study since they are not included in the list of the initial twelve POPs under the Stockholm Convention at the start of this study.

The fish, human milk and ash samples and standards 1A and 1B were distributed by MTM, whereas the sediment sample and standard 1C were distributed by IVM.

2.2 Methods used by participants

The participants were not restricted in the methodology used for the analysis of the target compounds in this first UNEP intercalibration study. Although it is for example advisable to analyse dioxin-like POPs (*i.e.,* PCDD, PCDF, dl-PCB) with gas chromatography (GC) -

high resolution mass spectrometry (HRMS) systems, also results from low resolution mass spectrometry (LRMS) instrumentation was accepted. The use of 'high resolution' capillary GC is considered mandatory to achieve the separation needed for an accurate determination of the analytes. The laboratories used their own sample extraction and clean-up protocols, spiking schemes and internal QA/QC. The reporting forms for the different classes of chemicals and the different matrices prepared in Microsoft EXCEL were used by all participating laboratories to submit their results.

For the analysis of the PCDD/PCDF and dl-PCB most laboratories used GC-HRMS systems but three laboratories used GC-LRMS systems. Also, the majority of the labs used three columns to clean-up the samples after extraction: a multi-layer silica, an alumina oxide (Alox) or Florisil column and a carbon-based column. Several labs used an automated clean-up system where these three columns are incorporated. A few GC-HRMS labs did not use all clean-up columns and for example the Alox or the carbon column was omitted from the sample clean-up procedure. The fly ash samples were often treated with acid before (warm) Soxhlet extraction or pressurized extraction systems (such as accelerated solvent extraction, pressurized liquid extraction or similar systems) using toluene or toluene based mixtures. Only one laboratory used dichloromethane as the extraction solvent. Also for the sediment sample, Soxhlet and pressurized extraction systems were used with toluene or dichloromethane/hexane mixtures.

The freeze-dried fish sample was extracted by pressurized extraction systems, Soxhlet or liquid/liquid extraction (*e.g.,* after KOH/ethanol decomposition of the sample). A large variety of extraction methods were used for the milk samples ranging from liquid/liquid to supercritical fluid extraction after mixing with an absorbent or pressurized extraction systems or Soxhlet extraction.

Only a limited number of labs analysed the seven indicator PCB in the ash samples using similar clean-up steps as for PCDD/PCDF and dl-PCB (Soxhlet, multilayer silica and or Alox) and detection using GC-HRMS. A variety of GC columns with different polarity or dimensions for optimal separation were used. One laboratory used a GC/ECD system for the indicator PCB in ash.

Methods to analyse the sediment for basic POPs did not show much variation: most laboratories used GC/LRMS and two labs used ECD. The marker PCB in the fish and the milk sample were extracted by liquid/liquid, Soxhlet and pressurized extraction systems and fat removal was achieved by multi-layer silica, gel permeation or concentrated sulphuric acid (H_2SO_4). Again both GC/ECD (two labs) and GC-HRMS was used for detection, surprisingly no data were acquired using a GC/LRMS system. For the submitted data it was not clear if the marker PCB were analysed together with the dl-PCB, as a separate fraction apart from the dioxin analysis or by applying a complete separate extraction and clean-up procedure.

The analytical procedures to analyse the pesticides varied widely from using HRGC/HRMS, HRGC/LRMS to GC/ECD to detect the target compounds. Again, in several cases it was not clear from the data if a combined or separate pesticide analyses was performed.

Surprisingly several labs used GC/HRMS to analyse the pesticides, if we assume that this was reported correctly (in the method information section of the report forms). This is an interesting development and seems to be characteristic in the Asian region where GC-HRMS capacity seems to be available in large numbers. For all samples, a wide variety of sample extraction and clean-up methods were used including Soxhlet, pressurized extraction systems, liquid/liquid, ultrasonic extraction, GPC, multilayer silica, alumina and Florisil.

2.3 Data Assessment

The data assessment was carried out according to the principles employed in the data assessment of the QUASIMEME proficiency testing organisation (www.quasimeme.org). All data received from the participants were entered into a database and assessed using a standard procedure to allow direct comparison between participants. The approach of the assessment is based on the standard, ISO 13528 (2005), the IUPAC International Harmonised Protocol for Proficiency Testing (Advanced Draft) by Thompson *et al.*. (2006). Additions or differences in the assessment from these standards are given or referred to in this report. However, the assigned value and the laboratory assessment using z-scores are based on the Cofino Model (Cofino *et al.*, 2000).

Comparison between the robust statistics method for calculation of a mean and the Cofino model continues to be made, and where there are any significant discrepancies between the two methods, further investigative analysis was undertaken. The Cofino model is generally able to separate the effects of the method on the results and provide a more reliable estimate of the measurement relating to the method. The standard, ISO 13528, includes statistics for proficiency testing schemes, and uses robust statistics as a basis for the assessment. However, it is generally acknowledged that robust statistics cannot cope with more than 10 % extreme values, particularly with a skewed distribution. The Cofino model is able to routinely cope with these types of distribution and provide the best estimate of the consensus value, which may be used as the assigned value.

The Cofino model has been developed for the routine QUASIMEME assessments. The Cofino model uses a Normal Distribution Assumption (NDA). The assigned value is based on the Cofino NDA model without any trimming of the data. This approach includes all data in the evaluation and no subjective truncation or trimming is made. This model has been further developed to include Left Censored Values (LCV)[1]. The development of these models has been fully documented and published (Cofino *et al.*, 2000; Cofino *et al.*, 2005; Wells *et al.*, 2004). An overview of the assessment with explanation and examples is given in the Assessment Rules for the Evaluation of the QUASIMEME LP Studies Data (Wells and Scurfield, 2004).

The details of the Cofino Model were provided elsewhere (Wells *et al.*, 2004; Wells and Scurfield, 2004) but in summary, the approach is as follows:

- All data included in the assessment
- No data trimmed or down weighted

[1] *Left Censored Values* is the correct nomenclature for “less than” values

- Assigned values (AV) based on Cofino NDA model
- All LCV are also included, provided certain criteria are met

2.3.1 Plots

The performance of the laboratories in this study is illustrated in the z-score histograms. Where the assigned value for an analyte is indicative, the values are plotted as their original reported concentrations. The rules for confirming whether the consensus value should be an assigned value or an indicative value are given in the Assessment Rules for the Evaluation of the QUASIMEME LP Studies Data (Wells and Scurfield, 2004) with relevant examples.

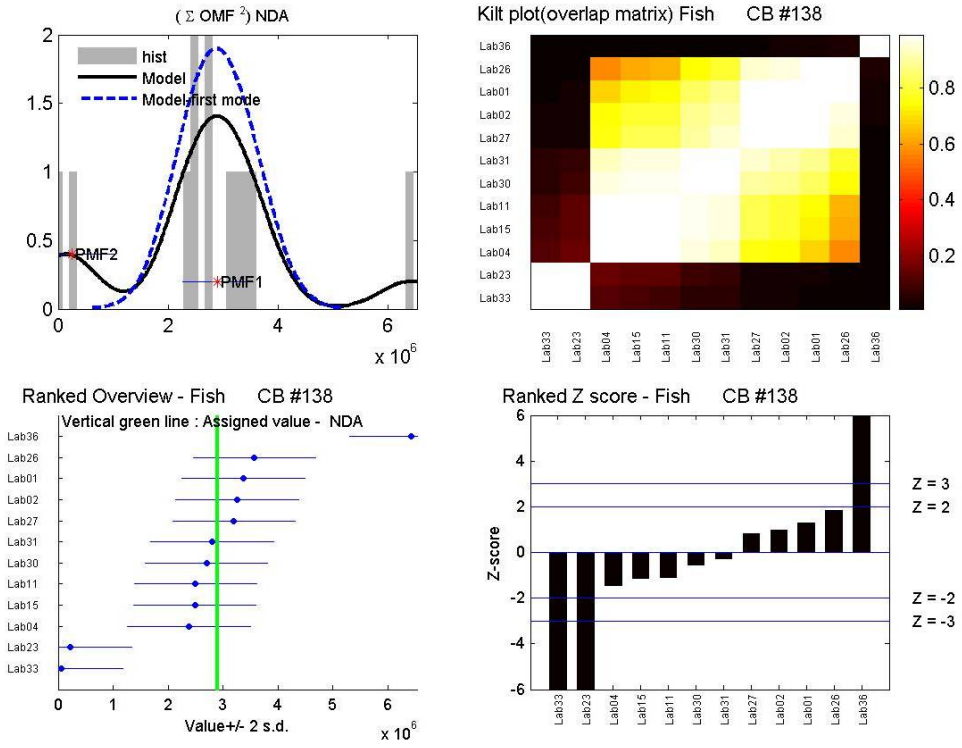


Figure 1. Graphical output of the Cofino Model statistics for PCB 138 in the fish sample.

Normally, four plots are given for each analyte (Figure 1). The upper left plot provides an impression of the probability density function (PDF) for all data (black) and for the first mode (blue dotted) (PMF1) of the data. Superimposed on these PDFs is a histogram of the individual measurements, given in grey. This plot shows the distribution of the data as a whole, and of the data in the main mode (PMF1) on which the assigned value is based.

The "Kilt Plot" (Overlap Matrix) (upper right plot) provides an overview of the degree of overlap of each pair of data. It gives a clear indication of the degree of homogeneity of the data. As a key, the white areas indicate maximum overlap of the PDFs and, therefore, highest agreement (an overlap of one implies that the two laboratories of the pair report exactly the same results), while the black area show the pairs in poor agreement.

The lower left plot is a ranked overview of all data with an error bar of ± 2 SD. The numerical values are given in blue and the left censored values are given in red.

The ranked z-score plot (lower right) is based on the mean of the data, which is normally also the assigned value. However, if there is any adjustment required to the assigned value as a result of the assessment, *e.g.,* use of the nominal concentration or a trimmed value, then the final z-score given in the z-score histograms will reflect these changes. In this study, no such adjustments are made and therefore, the z-score plot (lower right) is the definite plot for obtaining the individual lab z-scores.

2.3.2 The Assigned Values and indicative values

The Assigned Value (AV) is obtained from the main mode of the data using the Cofino Model (bleu dotted line in upper left panel in Figure 1), and is centered around the highest density of values. Unless otherwise stated, the assigned value is based on this consensus value of *all* data. Although *all* data are included in the assessment, those values that lie some distance from AV contribute less to the mean than values, which occur at or near the mean.

In some instances, it is not possible to set an AV, and an indicative value is given. No assessment of laboratory performance is given where an indicative value is set. An overview of the assessment, with explanation, decision flowcharts and examples, is given in the paper *Assessment Rules for the evaluation of the QUASIMEME Laboratory Performance Studies Data*, available on the QUASIMEME website, www.quasimeme.org. A summary of the categories is given below:

Category 1

For data with the number of numerical observations ≥ 7

An assigned value is based on the mean when ≥ 33 % of values have a z-score of $|z| < 2$. Where < 33 % of the data have $|z| < 2$ the value is indicative, *i.e.,* at least 33 % must be in good agreement.

Category 2

For data with the number of numerical observations > 3 and < 7

An assigned value is based on the mean when ≥ 70 % of values have a z-score of $|z| < 3$ and a minimum of 4 observations have $|z| < 2$. Otherwise, the value is indicative. *i.e.,* for small datasets, n > 3 and n < 7, there needs to be very good agreement and a maximum of one extreme value before an assigned value can be given.

Category 3

For data with the number of numerical observations < 4

No assigned value is given. Normally the median value is given as an indicative value.

Category 4

For data with the high Total Error% >100 % in combination with bad performance, no assigned value is given.

2.3.3 The z-score Assessment

A z-score (Thompson and Wood, 1993) is calculated for each participant's data for each matrix / analyte combination which is given an assigned value. The z-score is calculated as follows:

$$z\text{-score} = \frac{\text{Mean from Laboratory} - \text{Assigned Value}}{\text{Total Error}}$$

It is emphasized that in many interlaboratory studies the between-laboratory standard deviation obtained from the statistical evaluation of the study is used as 'total error' in the formula above. In the QUASIMEME data assessment, the total error is estimated independently taking the needs of present-day international monitoring programs as starting point. For each analyte in a particular matrix, a proportional error (PE) and a constant error (CE) have been defined. The total error depends on the magnitudes of these errors and on the assigned value:

$$\text{Total Error} = \frac{\text{Assigned Value} \times \text{Proportional Error (\%)}}{100} + 0.5 \times \text{Constant Error}$$

The values for PE and CE are set by the QUASIMEME Scientific Assessment Group and are monitored annually. The values are based on the following criteria:

- Consistency of the required standard of performance to enable participating laboratories to monitor their assessment over time.
- Achievable targets in relation to the current state of the art and the level of performance needed for national and international monitoring programmes.

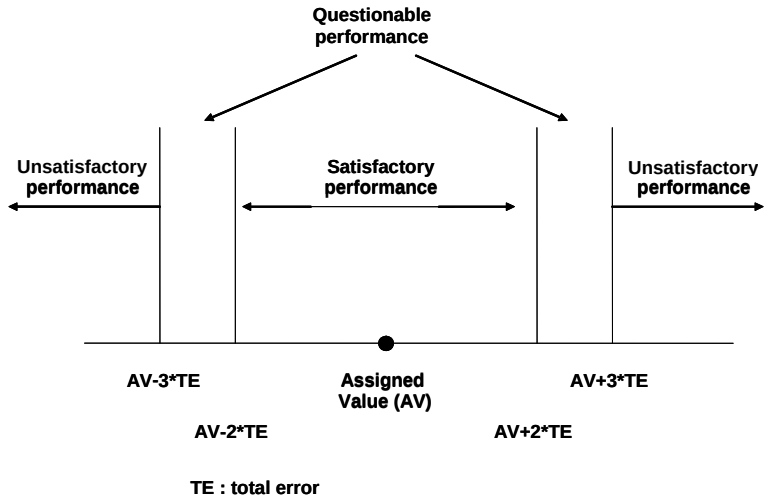
The assessment is based on ISO 43 as z-scores. The QUASIMEME model is designed to provide a consistent interpretation over the whole range of concentration of analytes provided, including an assessment where Left Censored Values (LCVs) are reported.

The PE in this study is set at 12.5 % for all matrices. This applies to all analytes. The CE has been set for each analyte or analyte group (*e.g.,* polychlorinated biphenyls). This value was initially set to reflect the limit of determination, but is at present more closely related to the overall laboratory performance. The magnitude of the CE is set to provide a constant assessment in terms of z-score regardless of concentration. Therefore, at low concentrations the level of accuracy required to obtain a satisfactory z-score is less stringent than at a high concentrations.

Following usual practices *e.g.,* ISO 43, the z-scores can be interpreted as follows for laboratories which take part in QUASIMEME to assure the quality of their data for use in international marine monitoring programmes:

$\|z\| < 2$	Satisfactory performance
$2 < \|z\| < 3$	Questionable performance
$\|z\| > 3$	Unsatisfactory performance

The following figure illustrates the interpretation of the z-scores:



$|z| > 6$ frequently points to gross errors (mistakes with units during reporting, calculation or dilution errors, and so on).

It is not possible to calculate a z-score for left censored values (LCVs). Quasimeme provides a simple quality criterion:

LCV/2 < (concentration corresponding to $|z|=3$): LCV consistent with assigned value

LCV/2 > (concentration corresponding to $|z|=3$): LCV inconsistent with assigned value, i.e. LCV reported by laboratory much higher than numerical values reported by other laboratories.

z score key:	S – Satisfactory
	Q – Questionable
	U – Unsatisfactory
LCV key:	C – Consistent
	I – Inconsistent
No data:	B - Blank

3. Results

The submitted results have been evaluated statistically and whenever the data met the requirements (as mentioned in chapter 2), an assigned value was established. z-scores were calculated based on the assigned value. Summary of the assigned values and the percentage of satisfactory to unsatisfactory z-scores are presented in the chapter "Results_1st UNEP Intercalibration Study – Asia Region" in tabular form.

4. Discussion

In total 38 laboratories from Asia, Europe and North-America participated in the present study. Of these, 34 laboratories submitted data on the test solution, 30 on the sediment, 20 on the human milk, 23 on the fish and 24 on fly ash.

Interlaboratory studies can provide some explanations of the relationship between the methods used and the results obtained. Unfortunately, poorly performing laboratories are often confronted with multiple difficulties, which makes it difficult to determine the exact sources of error. The present results and draft report were presented at a UNEP regional workshop in Hong Kong, SAR, China, in February 2010 where the Asian and all participating developing country laboratories were invited. During the meeting the participants evaluated their own performance and improvement of methodology. During a pilot project on capacity building, a few of the laboratories that participated in this study received training in analysis of POPs in biota and sediments. These laboratories also participated in a first interlaboratory study on PCB, PCDD/PCDF and OCP analysis.

Twenty-four laboratories originate from Asia (China, Malaysia, India and Vietnam) and 14 laboratories were based in OECD countries. The differences between reference laboratories in OECD countries and in developing countries were small. Of the fifteen laboratories performing poorly (< 60 % satisfactory z-scores), six were OECD countries. On the contrary, eight out of eleven best performing laboratories (> 90 % satisfactory z-scores) were non-OECD countries.

4.1 Methodological considerations

An overview of methods used by participants is presented in Chapter 2 and Appendix 2. It can be challenging to identify trends in an interlaboratory study dataset and to explain the underlying methodological causes. The number of laboratories submitting results for each group of contaminants, the concentration of the test material, and variations in the analytical methods used by the participants, are some of the factors that may blur the interpretation of the outcome (de Boer and Wells, 2006). Calculation and dilution errors are other factors that may impede the understanding of the data, and may be difficult to detect. Nonetheless, based on the results and previous experience with interlaboratory studies, several problems could be elucidated.

POPs in mother's milk and fish tissue are presented on a lipid weight basis. The interlaboratory comparison of lipid weight concentrations is vulnerable to interlaboratory variation in determination of lipid content (Miskiewicz and Gibbs, 1992). Furthermore, the combination of high lipid content and low concentrations tend to cause higher RSD values (de Boer and Wells, 2006).

Most of the laboratories that participated in the present study reported fairly consistent lipid contents, both in milk and fish. However, two laboratories reported values deviating one order of magnitude from the others. In an interlaboratory study on brominated flame retardants, the authors suggested that a high variability in lipid content occurred because the laboratories did not adapt standard analytical protocols to the new matrices (de Boer

and Wells, 2006). When organising an interlaboratory study precaution should be made to reduce the variability in determination of lipid content as it may hamper interlaboratory comparison using lipid based concentrations.

4.2 Laboratory performance on various matrices

As presented in the chapter "Results", there was a high variability between matrices. This can also be seen from the number of laboratories with satisfactory z-scores, as shown in Figure 2.

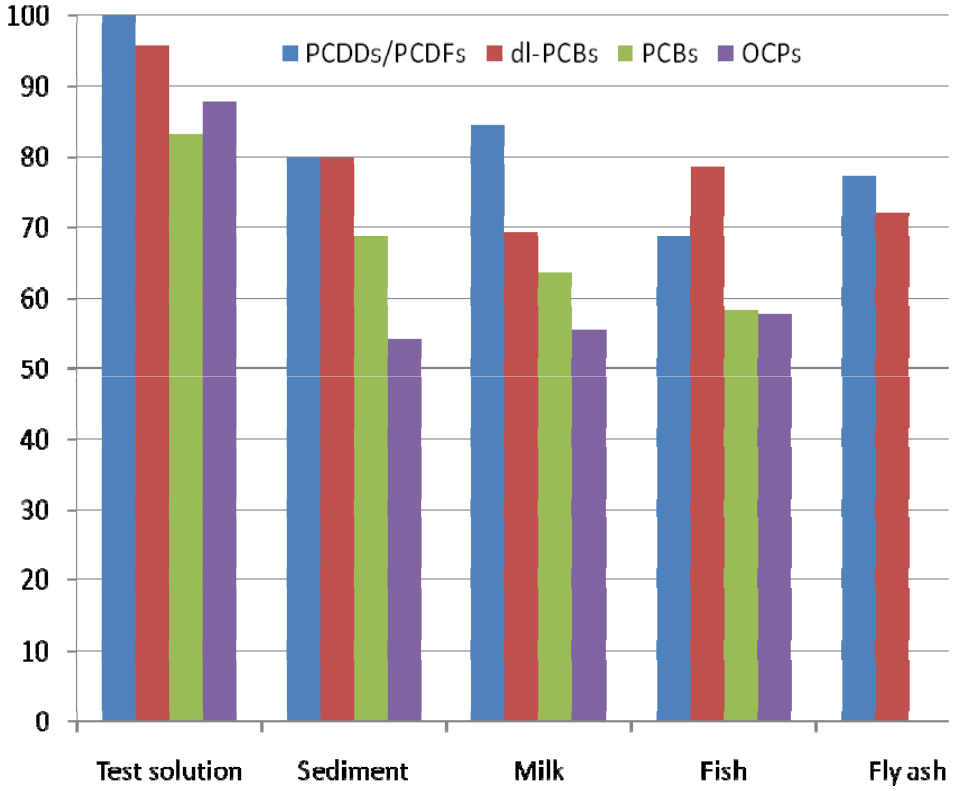


Figure 2. Percentage of laboratories with satisfactory z-scores (i.e., z< ± 2) for OCP, PCB, PCDD/PCDF and dl-PCB in the test solution, sediment, milk, fish and fly ash.

The overall performance of labs measuring the test solution (certified standard solution) was satisfactory. In average 90 % of laboratories reported an acceptable z-score (±2) for this matrix. In average, 67 % of all contaminants reported were within ± 10 % of target value, whereas 98 % were within ± 20 % of the target value. The RSD values for most of the contaminants were 10 %-20 %, thus suggesting that factors associated with calibration (standards, calibration curves, storage of standard solutions, etc.) are satisfactory.

Five percent of the test solution data was found unsatisfactory (z-score > 3). In comparison, between 59 %-64 % of laboratories reported acceptable z-scores for sediment, fish and milk. Between 10 %-22 % of the data on these matrices had an unsatisfactory z-score. For

the laboratories analysing fly ash, in average 75 % of the dl-PCB and PCDD/PCDF data was found acceptable. This shows that analysis of real matrices is more complex and results in a higher variety of the results.

In sediment, particular difficulties were seen for the lower chlorinated PCB and OCP (RSD > 200 %).

Better results were obtained for PCDD/PCDF. The PCDD/PCDF were present in lower concentrations (2-3 orders) compared to the PCB and OCP. However, due to the use of HRMS this did not cause any problems for the labs, on the contrary, the use of HRMS improved the results. The results for the standard solution were very good with an RSD of only 8 % for the total TEQ. The PCDD/PCDF results were also good for both the ash and sediment samples based on the total TEQ showing RSD of 24 % for both matrices. The PCDD/PCDF results for the total TEQ for the fish sample was satisfactory (33 %) taking all entries into account, after removing one extreme outlier this RSD improved to 18 % for the remaining 12 laboratories, which is exceptional good for this complex analysis. The same is applicable on the milk sample where the RSD for the total improved from 53 % for all 12 participating laboratories to 13 % after removing two outliers. This is all in agreement or in some cases better than reported in the literature when more than 15 years of 'dioxin' QA/QC studies were evaluated to establish 'fit for purpose' RSDs (van Bavel *et al.,* 2008).The RSD values for PCDD/PCDF and higher chlorinated PCB in milk were far better. In fly ash the PCDD/PCDF RSD values were found acceptable, whereas the individual dl-PCB showed larger variation due to some extreme outliers (RSD > 300).

OCP and PCB RSD values were in the range of 50 %-150 %. The fish sample was the matrix with the highest lipid content. In another matrix with a relatively high lipid content, milk, difficulties were particularly associated with OCP (RSD 100 %-200 %) and lower chlorinated PCB (100 %-300 %), suggesting errors associated with clean-up of the samples.

In average 59 % of the laboratories submitted test solution data, whereas 54 % submitted data on fly ash. Between 29 %-41 % of the laboratories submitted data on the other three matrices; sediment, milk and fish.

There was no clear indication of a "Horwitz trend" in the dataset, *i.e.,* lower concentrations inducing higher RSD values (Horwitz, 1980). Not even when PCDD, PCDF and dl-PCB were removed, *i.e.,* compounds analysed using internal standards, any Horwitz trend was detected. On the contrary, there appeared to be a greater bias for herring tissue and milk with relatively high concentrations, than for sediment and fly ash. A similar trend was identified in a previous interlaboratory study analysing sediment, herring and a test solution in seven developing countries (de Boer *et al.*, 2008). Milk and herring are more difficult to analyse, mainly due to their relatively high lipid content.

The good performance of most laboratories for the test solution suggests that extraction, clean-up and resolution and not instrumental sensitivity, are the main sources of error. Nevertheless, it should be noted that far less data was submitted for other matrices than for the test solution probably due to the difficulties associated with real samples. Some of the laboratories may simply not have been able to submit data on the matrices. There is also a possibility that laboratories did not report data they were not satisfied themselves.

The participating laboratories used in-house methods for sample preparation, clean-up, extraction and instrumental analysis. The participants were encouraged to use appropriate columns for the analyses. Due to lack of information related to each of the in-house procedures applied, and the numerous possible combinations of methods, the variability between laboratories is difficult to assess. De Boer and Wells (2006) observed that many laboratories, in spite of a better availability of analytical standards and ^{13}C-labelled standards, need extensive time in order to establish a new analytical method. It is not unlikely that some of the laboratories had not analysed some of the matrices included in the present interlaboratory study, and thus did not have sufficient time to adapt properly to the new methodology or, because of time constraints, chose to stick to methods they already were familiar with.

4.3 Contaminant group specific performance

The largest deviance from the assigned value was seen for OCP. on average only 62 % of the data had a satisfactory z-score, as compared to 79 % of dl-PCB and 82 % for PCDD/PCDF. There are numerous challenges that might have obstructed the analysis OCP in particular, from decomposition in the injector (dirty liner) to interfering substances and co-elution in combination with non selective ECD detection (de Boer and Wells, 1997). Possibly, some laboratories may have used sulphuric acid to remove lipids; however, this may disintegrate some OCP such as dieldrin (de Boer and Wells, 1997).

OCP like DDTs are easily degraded when the GC is not in the optimum condition (*i.e.,* dirty liner), resulting in inaccurate results. For indicator PCB, 69 % of the labs showed an acceptable z-score. In the QUASIMEME interlaboratory studies, the general performance of laboratories analysing POPs in sediment was found to be lower for OCP than PCB (de Boer and Wells, 1997). The authors noted that the vast majority of the participating laboratories were not able to the determine OCP levels with an acceptable accuracy. Even though this was thirteen years ago, it pinpoints some of the challenges encountered by several laboratories participating in the present study. The major problem with OCP analysis is in the GC/ECD analysis, which is in fact a compromise for a number of OCP. The ECD is not specific, the baseline is rather noisy, separation of early eluting compounds is not very good, and internal standards may not compensate for all losses. The use of GC/MS, even low resolution MS, together with ^{13}C labelled standards would improve this performance substantially, as is shown for the analysis of PCDD/PCDF, which are present at lower concentrations than the OCP.

4.4 Performance of specific laboratories

The performance of each individual laboratory was variable. A substantial part of the laboratories reported more than 70 % of submitted observations at an acceptable z-score (± 2). Four, six and eight laboratories reported even acceptable z-scores for 90 % of reported observations, for OCP, PCB and PCDD/PCDF/dl-PCB, respectively. Twelve, six and fifteen laboratories reported 70 %-89 % of observations at an acceptable z-score, for OCP, PCB and PCDD/PCDF/dl-PCB (on TEQ basis), respectively.

However, in most laboratories there is still need for further improvement of the analysis. A few laboratories had a very poor performance and require a substantial improvement in order to obtain a satisfactory analytical level.

The relative amount of submitted results with satisfactory z-scores (± 2) was between 33 % and 58 %. Four laboratories (labs 18, 23, 35 and 36) had a low performance for two of three contaminant groups, and ten laboratories (labs 3, 4, 5, 8, 15, 19, 25, 31, 33 and 37) scored low on one of the contaminant groups (all laboratories < 60 % of z-scores ± 2).

4.5 Comparison with other interlaboratory studies

The summary of RSDs for the five test matrices and the groups of POPs are shown in Table 1. The laboratory performance of PCB in the test solution was in general better (average RSD = 17 %) compared to a previous interlaboratory study including seven participants (average RSD = 57 %) (de Boer *et al.*, 2008), suggesting a better calibration of equipment. For OCP, the difference was slightly less, 19 % *vs.* 49 %, respectively.

In sediment and fish tissue, the results were in-line with the interlaboratory study from 2008. For the sediment test matrix, the laboratories participating in the present study had an average RSD value of 42 % and 117 % for PCB and OCP, respectively, in comparison to 150 % and 130 % in the study from 2008 (de Boer *et al.*, 2008). In herring tissue the results corresponded even more, the present laboratories reporting average RSD values of 83 % and 70 % for PCB and OCP respectively, in comparison to 65 % and 90 % as for de Boer *et al.* (2008).

The results are comparable to an interlaboratory study led by the International Atomic Energy Agency (IAEA), which reported RSD values between 30 % and 150 % for PCB and OCP in mussel homogenate (Villeneuve *et al.*, 2004). However, when compared to recent (mainly European) studies such as QUASIMEME, the present results are poorer (de Boer and Wells, 1997; and references herein).

Table 1. RSD and number of laboratories after removing obvious outliers

	Ash RSD	n	Sediment	n	Fish	n	Milk RSD	n	Standard	n
$TEQ_{PCDD/PCDF}$	18 %	22	19 %	19	18 %	13	16 %	11	8 %	28
TEQ_{PCB}	19 %	15	22 %	17	19 %	12	22 %	11	16 %	24
TEQ_{total}	19 %	17	17 %	16	18 %	12	13 %	11	8 %	23
PCB_7	*91 %**	9	*35 %**	16	*57 %**	12	14 %	10	12 %	22
Drins	-	-	*227 %**	4	40 %	8	29 %	10	15 %	22
Chordanes	-	-	99 %	8	26 %	8	46 %	9	17 %	19
DDTs	-	-	29 %	16	30 %	9	31 %	10	14 %	20
HCB	-	-	26 %	14	30 %	9	25 %	9	14 %	22
Mirex	-	-	22 %	5	29 %	9	29 %	9	9 %	18

** Italic, no outliers removed.*

During the UNEP workshop in Hong Kong (26-28 February 2010) the preliminary results were presented and discussed in detail, the UNEP RSD criteria of 12.5 % was lively dis-

cussed. This stringent criterion was set by UNEP to assure that the target decrease of POPs concentration in the core matrices can be monitored: The Guide for the Global Monitoring Plan (GMP) aims to show a 50 % decline in levels of the POPs over a ten year period. To demonstrate this decline is one of the decisive factors in the effectiveness evaluation of the Stockholm Convention (Article 16). This fixed criterion is somewhat different to the often used 'floating' RSD based on the analytical data of all 'expert' laboratories after the removal of obvious outliers.

To address this issue, the RSD of the data for the different samples and standard solutions after removing outliers by using the modified 'graphical consistency technique' as described in ISO 5725-2:1994 (E) are given in Table 1. These RSDs are often used to calculate z-scores based on the 'floating' RSD principle (van Bavel, 2008). Figure 3 to Figure 7 illustrate both the floating RSDs and the set 12.5% and 25% criteria are given in. Note that the figures from the 'raw' data files given in Appendix VI also contain obvious outliers, which in some cases results in extreme high RSDs (> 100 %). The corrected data after outlier removal is added to the 'raw' data in Appendix V.

As can be seen from Figure 3 (and a Table in the Results chapter), the RSDs for the total TEQ after outlier removal are very close or below 12.5 %, and corresponding z-scores are in most cases similar or below the z-scores calculated by using a floating RSD. Also for the total TEQ for the sediment sample, illustrated in Figure 4, the fish (18 %), the milk (13%) and ash (19 %) the differences are marginal.

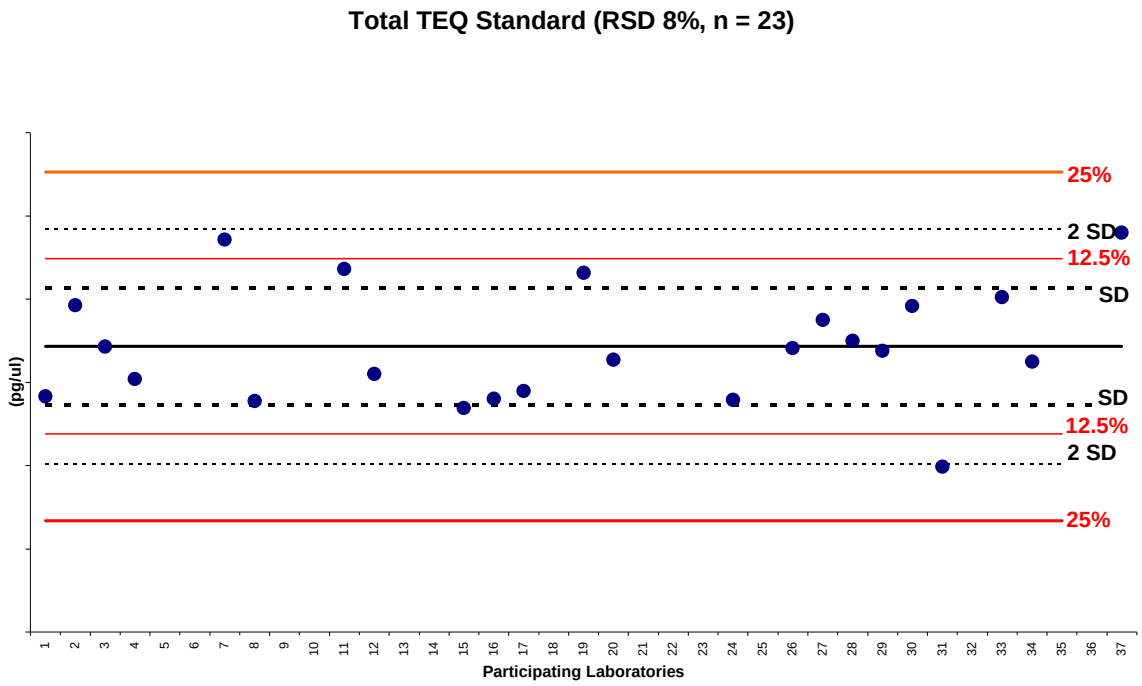


Figure 3. Results for the total TEQ in the standard solution. Dotted lines represent 1x the SD and 2x the RSD, the solid red lines represent the 12.5 % and 25 % UNEP criteria.

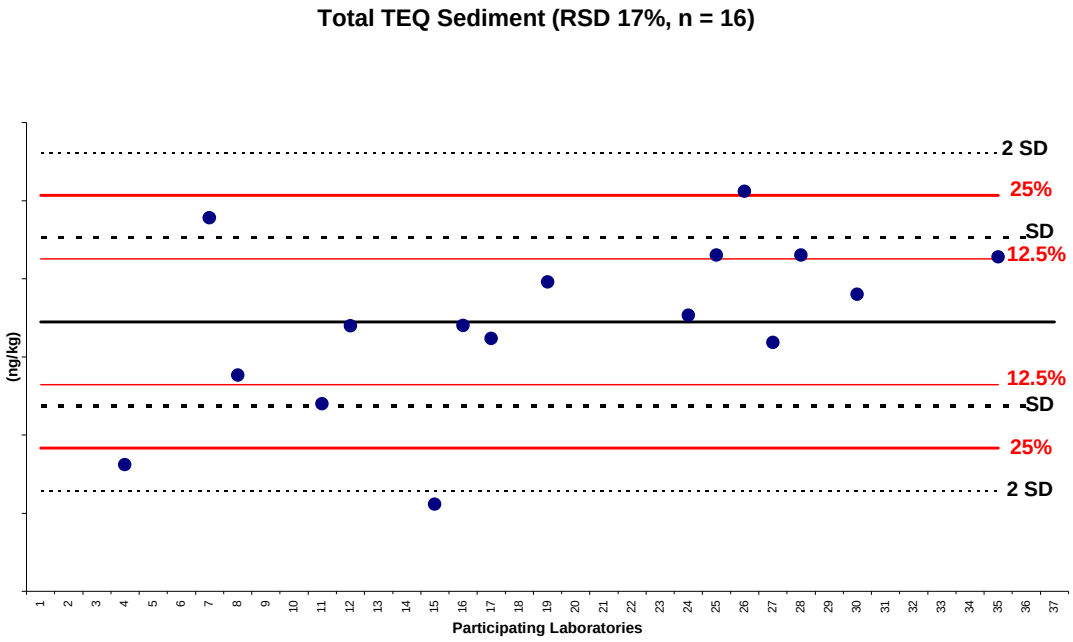


Figure 4. Results for the total TEQ in the sediment sample. Dotted lines represent 1x the SD and 2x the RSD, the solid red lines represent the 12.5 % and 25 % UNEP criteria.

For the PCB results, the milk (Figure 5) and the standard solution (12 %) are in line with UNEP's 12.5 % criteria. However, the RSDs for the results of the ash (91 %), the fish (57 %) and the sediment (35 %) are much larger. This difference is illustrated in Figure 5, where the results for the indicator PCB are given for the sediment sample.

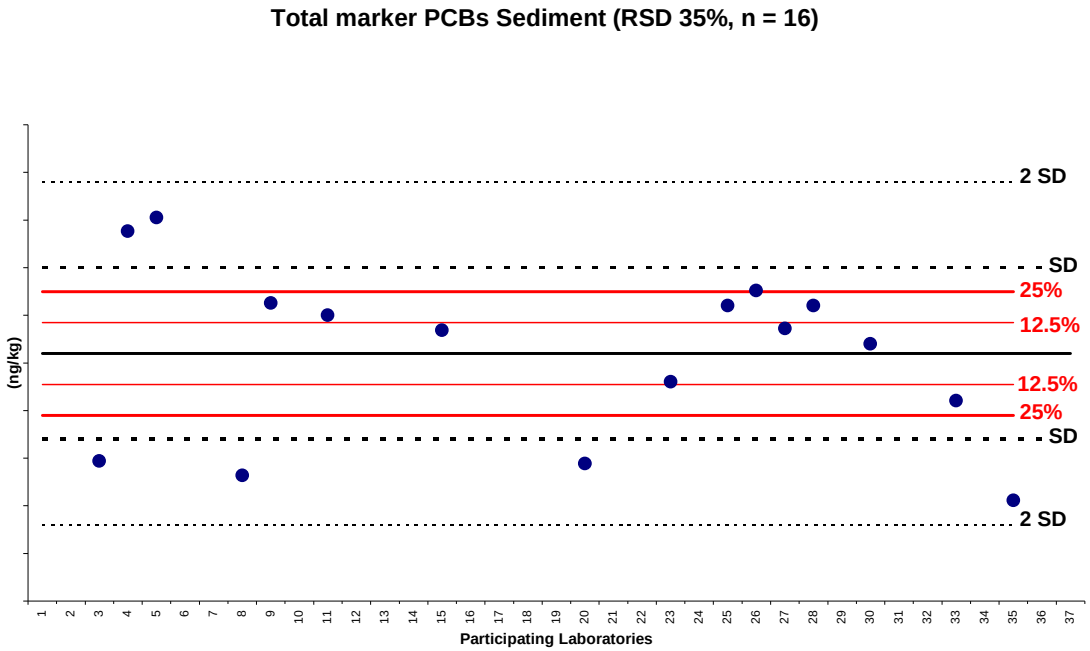


Figure 5. Results for the marker PCB in the sediment sample. Dotted lines represent 1x the SD and 2x the RSD, the solid red lines represent the 12.5 % and 25 % UNEP criteria.

Somewhat surprisingly, the results for the pesticides show much larger variation and larger RSDs even after outlier removal, both, for the individual compounds mirex and HCB, and the sum parameter for the chlordanes, DDTs and drins. The RSDs after outlier removal still varied from 22 % to 227 % for the sediment, fish and milk, and between 9 % and 17 % for the standard solution. The large variation in the pesticide data is illustrated for the sum of DDTs in both sediment and fish in Figure 6 and Figure 7, respectively.

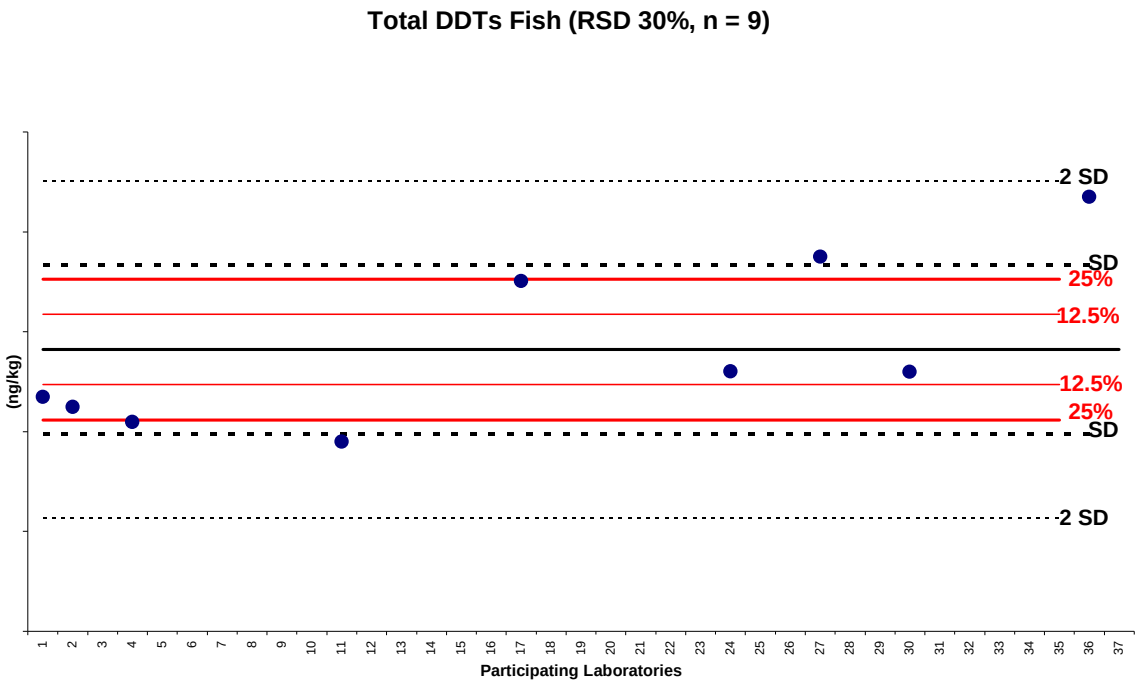


Figure 6. Results for the sum of PCB in the fish sample. Dotted lines represent 1x the SD and 2x the RSD, the red lines represent the 12.5 % and 25 % UNEP criteria.

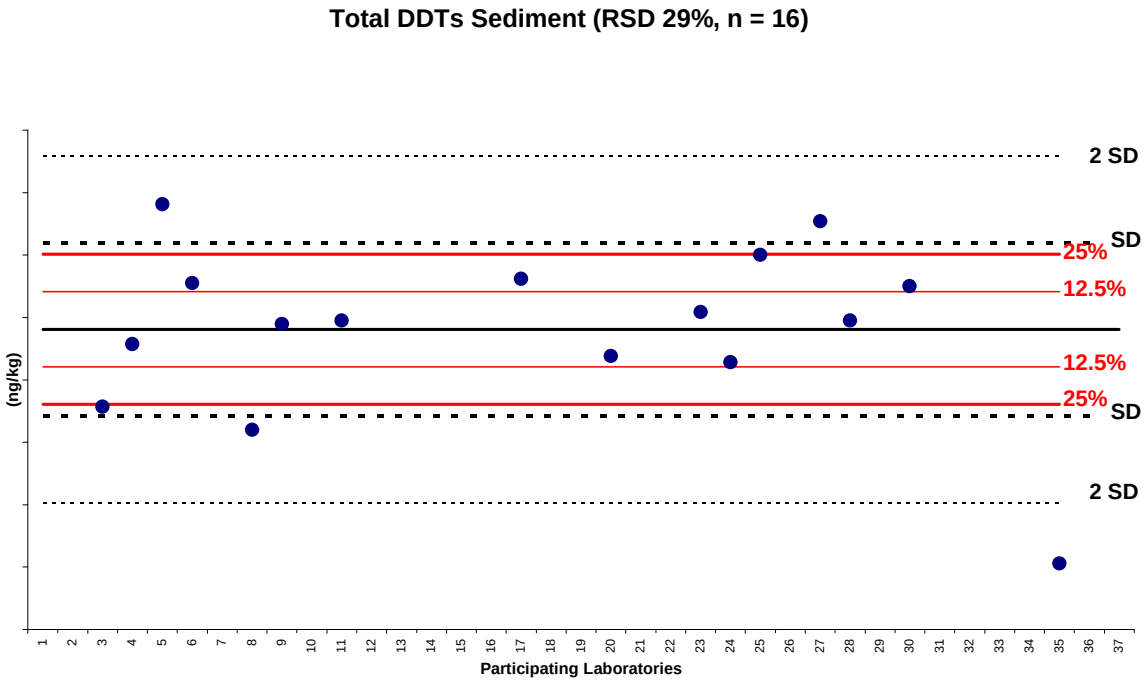


Figure 7. Results for the sum of PCB in the sediment sample

Especially when there is a large variation in the data set and outlier removal does not improve the RSDs or is not possible due to the distribution of the data, it is important to cal-

culate the assigned values as accurate as possible. This importance has been illustrated in section 2.3, where the Cofino statistical approach is explained. In Table 2, the assigned values calculated by Cofino statistics and outlier removal are given. Again, the difference between the two statistical approached is marginal except for some of the pesticides in fish sample. As illustrated in Figure 6, a limited number of data points and a skewed data distribution are responsible for this difference. The detailed data is included in the section "Results" as Table 9.

Table 2. Assigned values according to Cofino statistics and outlier removal (Intercal)

	Ash	Sediment	Fish	Milk	Standard
$TEQ_{PCDD/PCDF}$	-1%	1%	0%	3%	0%
TEQ_{PCB}	4%	-2%	-1%	-8%	0%
TEQ_{total}	1%	4%	0%	-5%	-1%
PCB		6%	9%	1%	-1%
Drins			3%	-3%	3%
Chlordanes			-7%		9%
DDTs		2%	-11%		6%
HCB		-3%	-11%	-8%	-1%
Mirex		2%	-6%	5%	2%

Taking into account that the aim of the QA/QC effort is that the data generated by the regional laboratories will be made available for the GMP and that within the GMP a decline of 50 % in levels over a time period of 10 years has to be established, the variation between qualifying laboratories should not exceed 2x 12.5 % = 25 %. Although it will be a challenge for all laboratories to achieve this, the data support that this is possible especially for PCDD/PCDF (as $TEQ_{PCDD/PCDF}$), dl-PCB (as TEQ_{PCB}). For the pesticides and the indicator PCB, more work is needed and it will be interesting to study the upcoming QA/QC studies and compare with the results in the other UN regions (especially Africa and GRULAC; the number of laboratories from CEE region is assumed to be small).

Considering the fact that this is the first phase of the first worldwide interlaboratory study on POPs, including 38 laboratories and the status and working conditions of many of the participating laboratories, the outcome is encouraging. However, comparing with some of the first interlaboratory studies on PCB and OCP in Europe, reporting CVs of 39 % and 41 % (both mean PCB) (Uthe *et al.*, 1988; Anon., 1993), the results presented here are weaker.

5. Conclusions

Analytical interlaboratory variability in POPs analysis is well documented (*e.g.* Mizikiewicz and Gibbs, 1992; de Boer and Wells, 1997; Holst and Müller, 2001). Although the present outcome is in line with a recent UNEP interlaboratory on POPs in developing countries (de Boer *et al.*, 2008), the results were in general poorer than interlaboratory data from Europe during the 1990s and early 2000 (*e.g.,* Rimkus *et al.*, 1993; Boekholt, 1993; de Boer *et al.*, 1996; Holst and Müller, 2001). The results for PCDD/PCDF and dl-PCB were good and in agreement with and in some cases better than previously reported for this complex analysis (van Bavel *et al.,* 2008).

An overall good performance on the test solution indicates that calibration is rather satisfactory at most laboratories. However, a substantial number of laboratories struggled with the analysis of 'real' matrices such as sediment and fish.

Poor performance was rather related to a variety of reasons than to one or two specific parts of the analysis. Laboratories were sometimes biased for certain samples only, sometimes for one or two contaminant groups and sometimes for all contaminants. Specific contaminants from the OCP group (*e.g.,* dieldrin and endrin) are vulnerable to degradation during extraction and clean-up as well as a dirty GC system. In addition, ECD detection is commonly used for detection of OCP and because of interferences, inaccurate results can easily be obtained. It is assumed that application of GC/MS systems would substantially improve the OCP results.

In general, the performance of OECD laboratories did not differ substantially from the performance of laboratories from developing countries, suggesting a fast development and improvement of POPs analysis in the latter group. Furthermore, the results emphasise the need for **all** laboratories to pay more attention to quality assurance (QA) and method development. Furthermore, it is imperative that authorities, management and others provide the resources necessary for an adequate QA-scheme in each laboratory. Regular, routine analyses instead of one-off projects would help to build up the required level of experience for this type of analysis.

6. Acknowledgements

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8. Appendices

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